

(12) **UK Patent Application** (19) **GB** (11) **2 303 860** (13) **A**

(43) Date of A Publication **05.03.1997**

(21) Application No **9615145.1**

(22) Date of Filing **19.07.1996**

(30) Priority Data

(31) **19526886**

(32) **22.07.1995**

(33) **DE**

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(51) INT CL⁶

C01B 3/32

(52) UK CL (Edition O)

C5E E101 E111 E126 E127

U1S S1360

(56) Documents Cited

US 4865624 A

(58) Field of Search

UK CL (Edition O) C5E EAP EAQ

INT CL⁶ C01B 3/32 3/38

Online: WPI, Claims, Japlo

(54) **Methanol reforming**

(57) A gas mixture to be reformed e.g. CH₃OH and H₂O, is passed into a catalyst containing reactor whose temperature controlled active length and/or inlet cross-section is set as a function of gas mixture throughput so that the residence period of the gas mixture remains constant in a feed side reactor section. The H₂ product gas obtained has a low CO content. Suitable reformer apparatus for the process includes a heating circuit in thermal contact with the reactor and means for the variable setting of the heating fluid flow rate actuable with respect to the gas mixture throughput. Alternatively, the apparatus may have a heating space which can be delimited by a piston arrangement or may comprise a plurality of reaction tubes which may be selectively blocked.

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Fig. 1

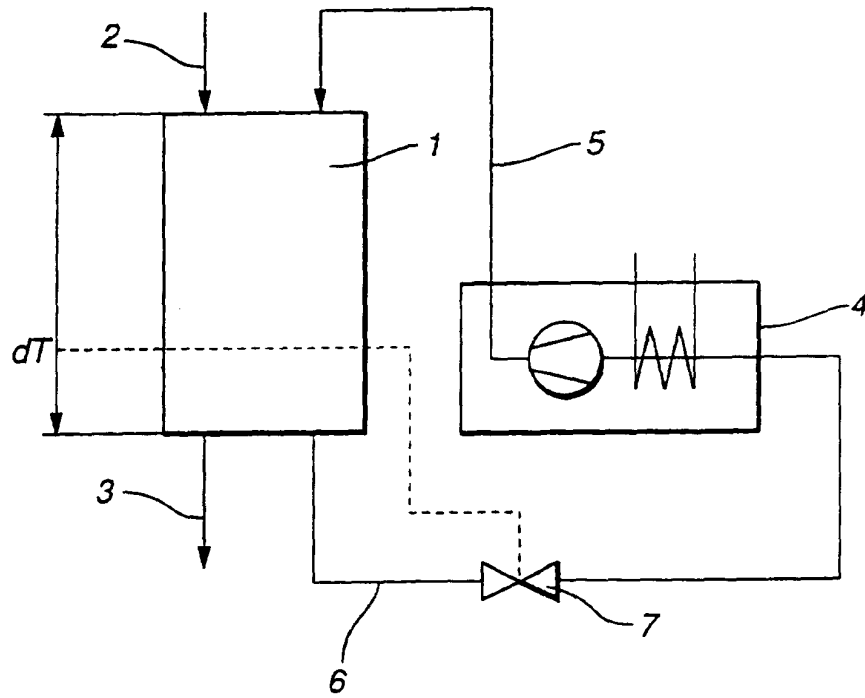


Fig. 2

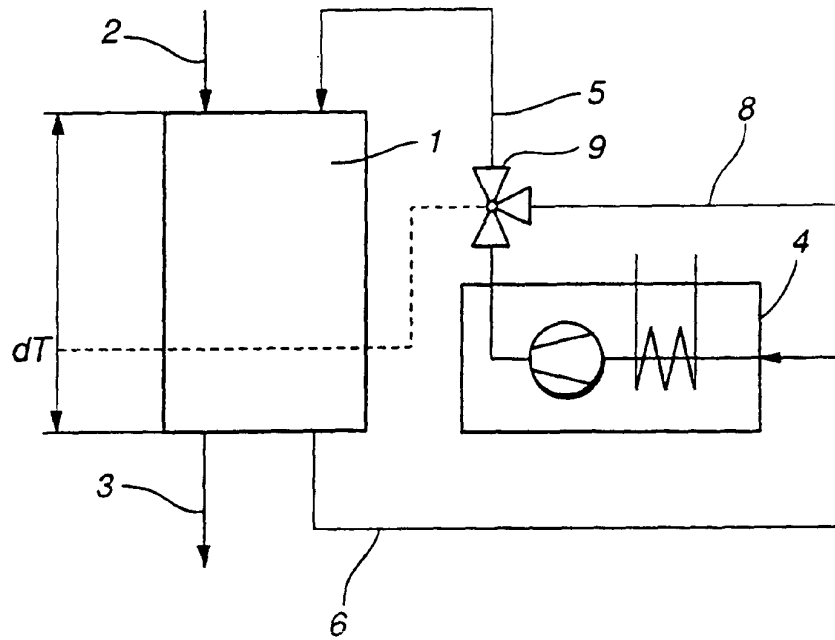


Fig. 3

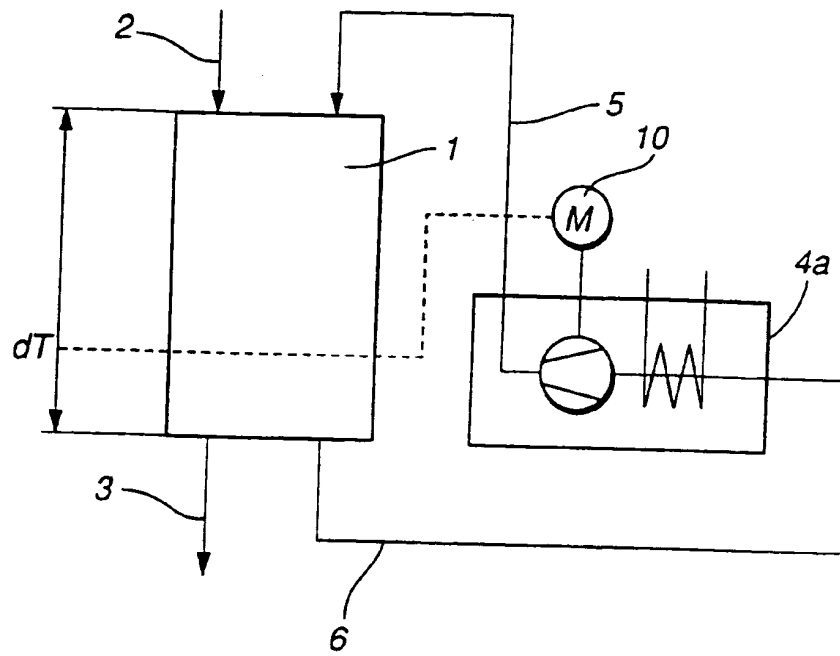


Fig. 4

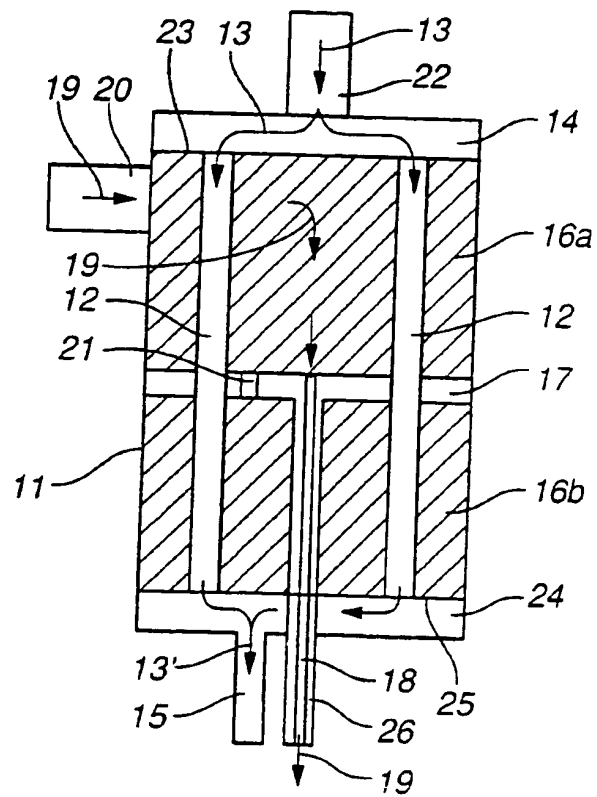
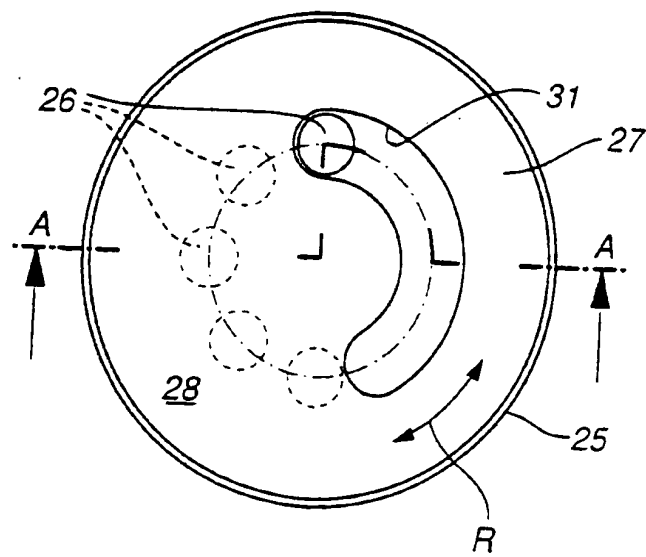
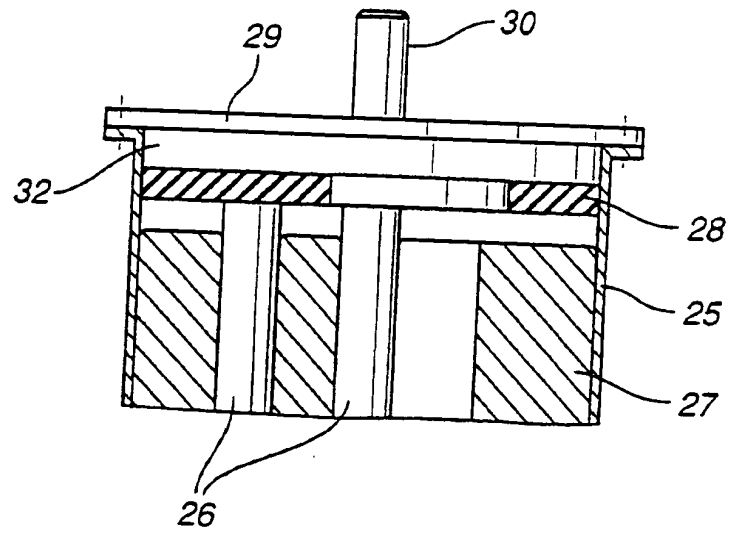


Fig. 5



Process and apparatus for methanol reforming

The invention relates to a process for methanol reforming, in which the gas mixture to be reformed is passed through a catalyst-containing reaction compartment, and to an apparatus suitable for carrying out this process.

A process of this type is used, in particular, for producing hydrogen gas. In this case, the methanol, for example together with water, is reacted in a hot-steam reformer to form hydrogen and carbon dioxide, the latter, together with the hydrogen, in turn being in a reaction equilibrium with water and carbon monoxide (CO. Methanol conversion rate and carbon monoxide production are therefore factors which are connected to one another via the, inter alia, temperature-dependent reaction equilibria, i.e., depending on the methanol conversion rate set by the process technology, a certain amount of carbon monoxide results when the currently known catalyst materials are used. In many cases the carbon monoxide, because of its toxicity for the environment and the catalyst materials used, is an undesirable by-product which must frequently be removed in a complex manner. Whereas for an industrial large-scale process, under steady-state conditions, an optimum operating point can still be maintained comparatively simply, under non-steady-state conditions, which means, principally, throughputs varying with time of the gas mixture to be reformed and associated changes in the catalyst space velocity in the reformer, operating phases may occur in which a relatively large amount of carbon monoxide is formed. Non-steady-state conditions of this type exist, for example, in non-steady-state applications of hydrogen as an energy source, such as for peak current generation and in motor vehicle drive units, in which one would wish to have recourse to methanol as a liquid source of hydrogen, in order not to require direct storage of hydrogen gas.

Since the methanol decomposition reaction proceeds more vigorously endothermically and the carbon monoxide

formation from carbon dioxide proceeds more feebly endothermically, to minimize the CO formation, it has already been repeatedly proposed to establish a temperature gradient along the reaction path of the gas mixture in the reaction compartment, in which the temperature decreases from the inlet side to the outlet side of the reaction compartment. Thus, in US Patent 4,865,624, a process and an apparatus for methanol reforming are proposed, in which, by means of suitable heating circuits, an inlet-side half of a catalyst-containing reaction compartment through which the gas mixture to be reformed can flow is kept at a higher temperature in a co-current flow process, and a subsequent outlet-side half is kept at a lower temperature in a counter-current flow process. This is said to promote the methanol conversion in the intake stage and the conversion of carbon monoxide into carbon dioxide in the exit stage. In a similar manner, the publication DE 41 93 026 T1 proposes the establishment of three temperature zones, situated one after the other, of approximately 300°C, approximately 275°C and approximately 225°C along the reaction path in a methanol reformer. In the laid-open application JP 63-50302(A, a stepwise temperature decrease in the reaction compartment of a methanol reformer along the gas mixture flow path to decrease CO formation is likewise specified. In all of these conventional processes, the parameters of the reforming reaction are preset independently of the respective instantaneous throughput of gas mixture to be reformed.

The present invention seeks to provide a process of the type mentioned at the outset and an apparatus suitable for carrying it out, by means of which methanol, even in the case of varying throughputs of gas mixture to be reformed, e.g. as a result of fluctuating hydrogen gas requirement, can be reformed in such a way that the reformat leaving the reaction compartment contains relatively little carbon monoxide.

According to the present invention there is

provided a process for methanol reforming, in which the gas mixture to be reformed is passed through a catalyst-containing reaction compartment, wherein the active length and/or the active inlet cross-section of an intake-side reaction compartment section which is temperature-controlled to achieve a high methanol conversion rate is set as a function of the respective throughput of gas mixture to be reformed in such a manner that the residence period of the gas mixture remains essentially constant in the intake-side reaction compartment section which is temperature-controlled for high methanol conversion.

The invention also provides apparatus for methanol reforming, having a catalyst-containing reaction compartment through which the gas mixture to be reformed can flow and a heating fluid circuit in thermal contact with the reaction compartment, wherein the apparatus is adapted to carry out the process referred to in the previous paragraph and includes a device for the variable setting of the heating fluid volumetric flow rate in the heating fluid circuit, which device is actuatable as a function of the throughput of gas mixture to be reformed.

In terms of the process, the active length of the intake-side reaction compartment section which is temperature-controlled to achieve a high methanol conversion rate and/or its active cross-section is set to be variable as a function of the particular throughput of gas mixture to be reformed in such a way that the residence period of the gas mixture to be reformed remains essentially constant in the reaction compartment section temperature-controlled to achieve a high methanol conversion rate. By this means, even under non-steady-state operating conditions, an optimum operating state with respect to minimum CO formation can be set in the methanol reformer.

Underlying this is the experimentally confirmed consideration that the methanol decomposition reaction on the one hand and the CO formation reaction on the other hand proceed at different speeds at different values of

parameters such as reaction compartment temperature and residence period in the reaction compartment of the gas mixture to be reformed, so that for each combination of reaction compartment temperature and residence period, an operating state optimum exists having high methanol conversion and low CO formation. By means of the process of the invention, the operating point of the methanol reforming can always be kept at this optimum operating state, as a function of the possibly fluctuating gas mixture throughput. Experimentally confirmed considerations of the reaction procedure indicate that, in the methanol reforming, the methanol is first reacted and carbon monoxide is only formed to an increasing extent in an exit-side part of the reactor path length no longer utilized for methanol conversion at low throughputs. By matching the active length of the intake-side reaction compartment section, which is temperature-controlled to achieve a high methanol conversion rate, to the respective instantaneous gas mixture throughput, it is therefore always possible by the present process to avoid such a downstream reaction compartment section which is heated to a higher temperature and to which virtually no methanol passes any more and which causes CO formation to an increased extent. Additionally, or alternatively, to this length matching, the residence period of the gas mixture to be reformed can be kept essentially constant, independently of the respective gas mixture throughput, in the intake-side reaction compartment section which is temperature-controlled to achieve a high methanol conversion rate by the fact that the active intake-side reaction compartment cross-section is changed as a function of the respective gas mixture throughput.

In a development of the process, the length of the intake-side reaction compartment section active for the methanol conversion is matched by the fact that the volumetric flow rate of the heating fluid used for the temperature control of the reaction compartment is set appropriately, the associated heating fluid circuit being

operated in co-current flow, i.e. the heating fluid flows in the area of thermal contact with the reaction compartment in parallel to the gas mixture to be converted. In this manner, the temperature gradient along the reaction compartment can be adjusted, and thus the length of the reaction compartment section can be adjusted which is temperature-controlled to achieve a high methanol conversion rate.

In an alternative development of the process, the length is matched by the fact that the length of the part of the heating fluid circuit in thermal contact with the reaction compartment is varied suitably.

In a further development of the process, the intake-side reaction compartment cross-section active for high methanol conversion is changed by the fact that reaction part-compartments which are separate from each other and arranged in parallel are optionally individually released or blocked.

By means of the apparatus, the volumetric flow rate of the heating fluid serving for the temperature control of the reforming reaction compartment can be altered in the associated heating fluid circuit via the device provided for this purpose, which enables the process according to Claim 2 to be carried out.

In a preferred embodiment of the apparatus, the heating fluid circuit for the temperature control of the reaction compartment contains a temperature-control compartment in thermal contact with the reaction compartment, which temperature-control compartment is delimited by a wall which can be displaced in parallel to the direction of flow of the gas mixture to be reformed. By displacing the wall, the length of the thermal contact between temperature-control compartment and reaction compartment may therefore be set and thus, e.g., the process according to Claim 3 may be carried out.

Another embodiment of the apparatus forms a tube-bundle reformer, in which a desired number of reactor tubes can be released or blocked, by which means, e.g., the

process according to Claim 4 can be carried out.

Preferred embodiments of the invention are shown in the drawings and are described below. In the drawings:

- Fig. 1 shows a block diagram of a methanol reformer having a heating fluid circuit equipped with a mainstream control valve,
- Fig. 2 shows a block diagram of a methanol reformer having a heating fluid circuit equipped with a bypass line and control valve,
- Fig. 3 shows a block diagram of a methanol reformer having a heating fluid circuit equipped with pump speed controller,
- Fig. 4 shows a cross-sectional view through a methanol reformer having an active temperature-controlled compartment of variable length and
- Fig. 5 shows, in the lower part, a plan view of the intake side of a tube-bundle reformer having an intake-side rotating opening disc and, in the upper part, a cross-sectional view of the intake-side area along the line A-A shown in the lower view.

The methanol reforming plant diagrammatically shown in Fig. 1 contains a reformer vessel 1, which includes a reaction compartment into which a methanol/steam mixture 2 can be introduced on the intake side in order to subject this to a hot-steam reforming reaction. In this case, the usual reaction conditions can essentially be set, which concerns, e.g., the pressure and the order of magnitude of the temperature of the reaction compartment and the choice of suitable catalyst materials in the reaction compartment. The construction of the reaction compartment can also be one of the structures known in tube-bundle reformers or plate reformers, without further details needing to be considered here. At the exit of the reaction compartment, the reformat 3 then exits, which essentially comprises hydrogen gas.

In order to ensure a maximum methanol conversion rate of more than 95% and, at the same time, to minimize the

high CO formation accompanying this, a temperature gradient dT is established, by means of a thermal oil heating circuit, over the length of the reaction compartment through which the methanol/steam mixture flows. The temperature gradient dT is designed in such a way that the reaction area of significant CO formation, which is situated downstream of the initial reaction area of high methanol conversion, is already at a comparatively low temperature level, so that the CO formation is suppressed. Depending on the load case, i.e. depending on the requirement of hydrogen gas produced, e.g. for a motor vehicle fuel cell downstream of the reformat exit 3 of the reformer vessel 1 and associated reformat gas production, the temperature gradient is set appropriately between the higher temperature at the reaction compartment intake side and the lower temperature at the reaction compartment exit side. For this purpose, the thermal oil heating circuit contains a heating and circulation unit 4 having a heating device and circulation pump. This unit 4 heats the thermal oil to the temperature desired at the inlet of the reformer vessel 1 and transports the thermal oil via a feed line 5 to a co-current flow heat exchanger unit, which is not shown in more detail, in the reformer vessel 1, which heat exchanger unit is in thermal contact with the reaction compartment, the thermal oil flowing through the heat exchanger in the same direction of flow as the methanol/steam mixture 2 to be reformed flows through the reaction compartment.

From the exit of the heat exchanger unit, a return line 6 of the heating circuit leads back to the heating and circulation unit 4, a control valve 7 which can be actuated via a pneumatic motor or the like being introduced into the return line 6. Since the reforming reaction proceeds endothermically, this removes heat from the thermal oil circulating through the heat exchanger unit, so that along the reaction path in the reformer vessel 1 the temperature gradient dT is formed, which also primarily depends on the throughput of methanol/steam mixture and on the volumetric

flow rate of the circulating thermal oil. The flow velocity of the methanol/steam mixture 2 to be reformed fluctuates in the reaction compartment as a function of the height of the instantaneous reformer loading, i.e. the instantaneous amount of reformat 3 removed, and thus the residence period of the methanol/steam mixture per unit length of the reaction compartment fluctuates. Accordingly, for a constant level of methanol conversion rate for a higher reformer loading, a higher active length of an intake-side reaction compartment section is necessary in which the methanol conversion essentially takes place.

This fact is taken into account in the reformer plant of Fig. 1 in that the control valve 7, by which the volumetric flow rate of the thermal oil circulating through the heating circuit can be controlled, may be adjusted as a function of the measured temperature gradient dT along the reaction compartment, as symbolized by the dashed line. At a higher load requirement, i.e. higher reformat withdrawal, the methanol/steam mixture 2 flows more rapidly through the reaction compartment, so that a greater intake-side reaction compartment length is necessary for effective methanol conversion and thus a shallower temperature gradient dT is necessary, an increased heat requirement resulting, moreover, owing to the endothermic reforming reaction. The excessively rapid temperature decrease which threaten for this reason in the intake-side reaction compartment section is then counteracted by the fact that the control valve 7 is further opened, so that the volumetric flow rate increases in the thermal-oil heating circuit. By this means, the increased heat requirement for the reforming reaction can be covered and, at the same time, because of the higher flow velocity of the thermal oil in the heat exchanger as well, the intake-side reaction compartment section can be appropriately extended, so that its length and temperature are just sufficient to convert the methanol at the highest possible rate. If, in contrast, the reformer loading falls, the methanol/steam mixture throughput decreases and the

temperature gradient dT thus tends to decrease. This actuates the control valve 7 in the closing direction, so that the thermal-oil volumetric flow rate in the heating circuit and, accordingly, the active length of the intake-side reaction compartment section serving for the methanol conversion decrease. The temperature gradient dT over the reaction compartment length can therefore be set appropriately as a function of reformer loading and accompanying reformat gas production in this reforming plant in such a manner that, independently of the respective throughput of methanol/steam mixture, an essentially constant residence period of the same is always maintained in an intake-side reaction compartment section which is heated to a higher temperature and is suitable for high methanol conversion, without decreased methanol conversion occurring because of an insufficient length of this section or increased CO formation occurring because of an excessive length of this section.

Fig. 2 shows a reformer plant whose structure and function essentially correspond to those of Fig. 1, in this respect, the same reference numbers being used and reference being made to the above description of Fig. 1. The plant of Fig. 2 is modified with respect to that of Fig. 1 to the extent that a by-pass line 8 bypassing the heating and circulation unit 4 and a three-way valve 9 are arranged, instead of a control valve in the return line 6 of the thermal-oil heating circuit. The by-pass line 8 is brought on the one side to the three-way valve 9 and the exit line of the heating and circulation unit 4 is brought on the other side to the three-way valve 9. The three-way valve feeds an adjustable mixing proportion from the two feed lines into the thermal-oil feed line 5 to the co-current flow heat exchanger unit in the reformer vessel 1. As is indicated by a dashed line, this three-way valve 9 is in turn triggered by the measured temperature difference dT between the intake and exit side of the reaction compartment in the reformer vessel 1. Since this means a control of the

volumetric flow rate of freshly heated thermal oil into the heat exchanger, the thermal-oil heating circuit built up in this way is functionally completely equivalent to that of Fig. 1, so that here also the intake-side reaction compartment length active for the methanol conversion can be matched to the reformer loading via the setting of a suitable temperature gradient dT by appropriate control of the thermal-oil volumetric flow rate in the heat exchanger.

A second variant of the reformer plant of Fig. 1 is shown in Fig. 3, again functionally identical parts being provided with the same reference numbers. In this methanol reformer also, control of volumetric flow rate of thermal oil circulating in a heating fluid circuit is provided to achieve a desired temperature gradient between the intake and exit side of the reaction compartment in the reformer vessel 1. Instead of the volumetric flow rate of the thermal oil being controlled via an actuatable control valve in the return line 6 of the heating circuit, as in the reformer of Fig. 1, in this reformer, the volumetric flow rate of the thermal oil is controlled by changing the pump speed of the pump provided in a circulation unit 4a, which is modified in this respect, of the heating circuit. For this purpose, a pump speed control element 10 is connected to this pump, which in turn can be actuated with respect to the measured temperature gradient dT by the control signal, as symbolized by the dashed line. If there is a higher requirement for reformat 3 and thus a higher throughput of gas mixture 2 to be reformed, the pump speed is increased, so that the thermal oil flows more rapidly through the heat exchanger in the reformer vessel 1 and a greater effective length of the reaction compartment is kept at an elevated temperature for high methanol conversion. Correspondingly, the pump speed is decreased, if the reformer loading becomes smaller.

Whereas, in the three methanol reformers described above, the temperature gradient dT over the reaction compartment length is matched to the reformer loading by controlling the rate of throughput of heated thermal oil, in

Fig. 4, a methanol reformer unit is shown in which the length of the intake-side reaction compartment section which is temperature-controlled for high methanol conversion can be set by changing the length of the thermal contact area between the influent thermal oil at elevated temperature and the reaction compartment. The methanol reformer shown is designed as a so-called tube-bundle reformer, in which the reaction compartment comprises a plurality of reaction tubes 12 arranged parallel to one another. The tubes 12 are situated within a reformer vessel 11, to one end of which is fed, via an inlet 22, the gas mixture 13 to be reformed, the incoming gas mixture 13 being distributed onto the various reaction compartment tubes 12 via an intake-side distributor gas space 14 of the reformer vessel 11. The reformat 13' exiting on the other side of the tubes 12 passes into an exit-side gas collection space 24 of the reformer vessel 11, from where it leaves the reformer vessel 11 via an outlet 15. Towards the interior of the reformer vessel 11, the distributor gas space 14 and the gas collection space 24 are each delimited by a partition 23, 25. The space between these two partitions 23, 25 in the reformer vessel 11 and outside the reformer tubes 12 forms a space filled with thermal oil which is partitioned into an intake-side half 16a and an exit-side half 16b by means of an axially slidable piston 17 having a moulded-on piston rod 26 leading out axially from the reformer vessel 11.

The thermal oil 19 serving as heating fluid enters via an inlet 20 on the reformer vessel 11 into the intake-side oil space half 16a. From there, it exits again from the reformer vessel 11 via a channel 18 which is introduced into the piston 17 and the piston rod 26 so as to run axially. The remaining components of the thermal-oil heating circuit are not shown here and can be implemented, e.g., in one of the ways shown in Figs. 1 to 3. Since, consequently, the active thermal oil circulation 19 does not lead through the exit-side oil space half 16b, the thermal oil situated there and, therefore, the exit-side reaction compartment section

enclosed by this, assumes a lower ambient temperature, whereas the actively circulating thermal oil 19 keeps the intake-side oil compartment half 16a and thus a corresponding intake-side reaction compartment length at an elevated temperature. By axially sliding the piston 17 through the reaction compartment 12 as a function of the gas mixture throughput, the length of the intake-side reaction compartment section which is temperature-controlled for high methanol conversion can therefore be set to the respective appropriate length at which the most complete methanol conversion just occurs, but no noticeable CO formation occurs. A narrow connection channel 21 between the intake-side and exit-side oil space halves 16a, 16b enables the thermal oil transfer, which is necessary for the sliding of the piston 17, from the diminishing to the enlarging oil space half, without causing a significant heat transfer from the intake-side 16a to the exit-side oil space half 16b.

In the methanol reformer of Fig. 5 shown in an upper area with its reformer vessel 25, the methanol-converting reaction compartment zone is matched to the reformat demand by changing the active intake-side reaction compartment cross-section in such a manner that, independently of the instantaneous gas mixture throughput, an approximately constant gas mixture flow velocity is maintained in each case. Accordingly, the residence period of the gas mixture in an intake-side reaction compartment section which is temperature-controlled for high methanol conversion is kept essentially constant. The methanol reformer is in turn designed as a tube-bundle reformer and contains six reaction tubes 26. These are arranged in parallel to one another at an equidistant spacing in such a manner that their longitudinal axes lie on a collective cylindrical half shell, as is to be seen in the plan view of the lower half of the figure. The reformer vessel 25 is closed on the intake-side by a cover 29 into which an inlet 30 for the gas mixture to be reformed is introduced. This inlet 30 opens out into an intake-side distributor gas space

32. Towards the interior of the reformer vessel 25, the distributor gas space 32 is delimited by a rotatably mounted rotating opening disc 28, into which is introduced a curved recess 31 in such a way that, by rotating the rotating opening disc 28 clockwise or anticlockwise, as indicated by the double arrow R, optionally all six or only some of the end inlet orifices of the reaction tubes 26 lie in this curved recess area 31. Gas mixture can flow into each of those reaction tubes whose inlet orifices lie in the recessed curve area 31 of the rotating opening disc 28, whereas the remaining reaction tubes are covered on the inlet side by the rotating opening disc 28. By rotating the rotating opening disc 28, therefore, the number of reaction tubes through which the gas mixture to be reformed actively flows may be set. In the situation shown in Fig. 5, e.g., just one of the six reaction tubes 26 is open.

In this manner, the entire active reaction compartment cross-section can be matched to the respective existing gas mixture throughput. At a higher reformer loading, i.e. higher throughput, more reaction tubes are opened, whereas at lower reformer load, more reaction tubes are closed. Suitable control of the rotating opening disc 28 ensures that, independently of the fluctuating gas mixture throughput, the flow velocity of the gas mixture to be reformed remains roughly constant in the reaction tubes 26. It is thus not necessary to change the length of the intake-side reaction compartment zone which is temperature-controlled for a high methanol conversion rate, if the reformer loading changes, for which reason, in this reformer, the tube bundle 26 is enclosed on a fixed length by a thermal-oil heating space 27, which is only shown in its reformer-intake-side part, and into which heated, circulating thermal oil enters, analogously to the above-described examples. The residence period of the gas mixture in the intake-side reaction compartment section surrounded by the heating space 27, which remains constant because of the constant flow velocity, independently of the respective

instantaneous gas mixture throughput, ensures, even in the case of fluctuating reactor load, that the intake-side reaction compartment length which is heated to a higher temperature always corresponds to the length which is required for high methanol conversion, without an excessive length occurring, which would cause higher CO formation.

Obviously, in addition to the above-described examples, further modifications can be implemented in the context of the invention specified by the claims, in particular, instead of the described tube-bundle reformers having an exit side opposite to the inlet side, U-tube bundle reformers or plate reformers can also be used. Furthermore, in addition to the use of thermal oil as heating fluid, which is described in the illustrative embodiments, any other suitable heat transport medium in liquid and/or gaseous form can also be used. Finally, it is also possible to provide direct heating using a burner or a catalytic burner.

Claims

1. A process for methanol reforming, in which the gas mixture to be reformed is passed through a catalyst-containing reaction compartment, wherein the active length and/or the active inlet cross-section of an intake-side reaction compartment section which is temperature-controlled to achieve a high methanol conversion rate is set as a function of the respective throughput of gas mixture to be reformed in such a manner that the residence period of the gas mixture remains essentially constant in the intake-side reaction compartment section which is temperature-controlled for high methanol conversion.

2. A process according to Claim 1, wherein the active length of the intake-side reaction compartment section which is temperature-controlled for high methanol conversion is set by varying the temperature gradient produced by a heating fluid circuit in the reaction compartment by means of controlling the volumetric flow rate of the heating fluid as a function of the gas mixture throughput.

3. A process according to Claim 1, wherein the active length of the intake-side reaction compartment section which is temperature-controlled for high methanol conversion is set by varying the length of the part of the heating fluid circuit in thermal contact with the reaction compartment as a function of the gas mixture throughput.

4. A process according to any one of Claims 1 to 3, wherein the active intake-side reaction compartment cross-section is set by opening or blocking reaction part-compartments which are arranged in parallel separately from one another.

5. Apparatus for methanol reforming, having a catalyst-containing reaction compartment through which the

gas mixture to be reformed can flow and a heating fluid circuit in thermal contact with the reaction compartment, wherein the apparatus is adapted to carry out the process according to Claim 1 or 2 and includes a device for the variable setting of the heating fluid volumetric flow rate in the heating fluid circuit, which device is actuatable as a function of the throughput of gas mixture to be reformed.

6. Apparatus for methanol reforming, having a catalyst-containing reaction compartment through which the gas mixture to be reformed can flow and a heating fluid circuit in thermal contact with the reaction compartment, wherein the apparatus is adapted to carry out the process according to Claim 1 or 3 and includes a heating space which is in thermal contact with an intake-side reaction compartment section which is temperature-controlled for high methanol conversion and which heating space is delimited by a wall which can be slid in parallel to the direction of flow of the gas mixture to be reformed.

7. Apparatus for methanol reforming, having a catalyst-containing reaction compartment, through which the gas mixture to be reformed can flow, in the form of a tube bundle comprising a plurality of reactor tubes arranged in parallel, wherein the apparatus is adapted to carry out the process according to Claim 1 or 4, and includes a device for the optional, intake-side opening or blocking of a variable number of reactor tubes.

8. A process for methanol reforming, substantially as described herein with reference to, and as illustrated in, the accompanying drawings.

9. Apparatus adapted to carry out the process as claimed in claim 1 and substantially as described herein with reference to, and as illustrated in, the accompanying drawings.



Application No: GB 9615145.1
Claims searched: 1 - 9

Examiner: Michael R. Wendt
Date of search: 28 November 1996

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK CI (Ed.O): C5E (EAP, EAQ)

Int CI (Ed.6): C01B 3/32, 3/38

Other: Online: WPI, Claims, Japio

Documents considered to be relevant:

Category	Identity of document and relevant passage	Relevant to claims
X	US 4865624 (NIPPON SANZO KK) e.g. see figure 1 & Column 5 line 27 - Column 6 line 44.	1 & 5

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.